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Influence of Thermocycling on the Bond Strength of Polyetheretherketone and Polymethyl Methacrylate to Indirect Composite: An In-vitro Analysis

MEGHNA BUDATI[1], KEERTHI SASANKA[2]

ABSTRACT

Introduction: Polymethyl Methacrylate (PMMA) has been used in dentistry for a long time due to its excellent aesthetics and ease of use. Polyether Ether Ketone (PEEK) has garnered attention for its potential in dental applications because of its remarkable mechanical properties and biocompatibility.

Aim: To examine the Shear Bond Strength (SBS) of indirect composite resin when bonded to PEEK and PMMA, with assessments conducted before and after thermocycling.

Materials and Methods: This in-vitro study was conducted at the Department of Prosthodontics and Implantology, Saveetha Dental College and Hospitals, Chennai, Tamil Nadu, India, from April 2023 to May 2023. The study included 44 samples, grouped into two: Group 1, consisting of 22 PMMA samples, and Group 2, comprising 22 PEEK samples. A total of 44 indirect composite samples were prepared using clear silicone moulds. The Ceresin Shofu bonding agent was used for bonding the PMMA and PEEK to the indirect composite and was light-cured. Subsequently, the two groups were further divided into four subgroups: Group 1A, with 11 PMMA samples subjected to the thermocycling process, and Group 1B, with 11 PMMA samples tested without thermocycling; Group 2A, consisting of 11 PEEK samples tested with the thermocycling process, and Group 2B, comprising 11 PEEK samples tested without thermocycling. The specimens underwent SBS testing after an immersion in distilled water for 24 hours at 37°C. SBS was measured using a universal testing machine, and statistical analysis was performed using parametric independent sample t-tests (α=0.05).

Results: The study demonstrated a statistically significant difference in SBS for composites bonded to PMMA, which exhibited higher bond strength, with values of (7.922±0.285) MPa before thermocycling and (7.270±0.2694) MPa after thermocycling, revealing a significant difference ($p<0.005$). In contrast, the SBS for composite bonding to PEEK was slightly lower, with mean values of (5.520±0.341) MPa before thermocycling and (4.486±0.232) MPa afterwards ($p<0.01$).

Conclusion: The SBS of PMMA bonded to indirect composite is higher than that of PEEK. Furthermore, it was noted that aging resulted in a reduction of bond strength for both PMMA and PEEK when bonded to indirect composite.

Keywords: Aesthetics Bonding agent, Dental biomaterials, Shear bond strength, Universal testing machine

INTRODUCTION

The PMMA has been widely used in dentistry for interim restorations, customised trays, denture bases and temporary restorations due to its desirable attributes, including ease of polishing, repairability, flexibility and cost-effectiveness [1]. However, PMMA has inherent limitations such as fragility, reduced mechanical properties, limited antibacterial efficacy and polymerisation shrinkage, which can hinder its long-term use [2]. Although PMMA remains the material of choice for interim repairs, its mechanical properties are insufficient for prolonged use as a durable temporary restorative material [3,4].

The PEEK has gained attention in dental applications due to its excellent mechanical properties, biocompatibility and elasticity similar to bone [5]. PEEK exhibits a high melting point and an 80% compressive strength, attributed to its enhanced glass solidification and higher ketone content within the polymer chain [6,7]. Despite these advantages, PEEK has aesthetic limitations due to its grayish hue and low translucency, necessitating the application of veneering layers or resin composites to achieve satisfactory aesthetics [8,9].

Advancements in adhesive techniques, surface pretreatments and newer generations of primers and composite resins have improved the bonding efficacy of indirect composites. Indirect composite restorations are preferred for long-term temporary use due to their enhanced accuracy, durability and aesthetic properties, as they are fabricated extra-orally under controlled conditions [10].

The bond strength between PEEK and indirect composites has been studied in comparison with zirconia copings, showing that zirconia-composite bonding tends to be stronger due to the presence of reactive sites and increased surface energy [11]. Additionally, the enhancement of surface roughness over time, due to microcrack formation and/or irregularities, can aid additional mechanical interlocking between the zirconia and the composite, thereby boosting bond strength [12].

Previous studies have assessed the bonding effectiveness of PEEK, with findings suggesting its bonding capacity is comparable to zirconia when appropriate pretreatments and primers are utilised [13]. Research on PMMA bonding to composites indicates that composite resin modifications and denture teeth repair have traditionally been performed using chairside techniques, with adhesion mechanisms involving molecular bonding, mechanical interlocking and thermodynamic adhesion [14,15]. Surface roughening has been identified as a key factor in enhancing adhesion by increasing the surface area for mechanical interlocking and molecular interactions [16]. Roughening the surface can enhance adhesion and involves augmenting the surface area for mechanical interlocking and molecular interactions. Close contact between two surfaces is necessary for the molecular bonding mechanism, which enables intermolecular interactions between the adhesive and the substrate through chemical reactions [17].

Thermocycling is widely recognised as an effective method for simulating intraoral ageing, ensuring uniform stress distribution and providing insight into the long-term durability of bonded materials [7,18,19]. Given the increasing use of PEEK and PMMA in dental applications, understanding their bonding characteristics with indirect composites under thermal ageing conditions is critical [20]. The present study aimed to investigate the SBS of indirect composite resin bonded to PEEK and PMMA and to compare the bond strength before and after thermocycling. The null hypothesis states that there is no significant difference in SBS between indirect composite resin bonded to PMMA and PEEK before and after thermocycling.

MATERIALS AND METHODS

The present in-vitro study was conducted at the Department of Prosthodontics and Implantology, Saveetha Dental College and Hospitals, Chennai, Tamil Nadu, India, from April 2023 to May 2023, following approval from the Institutional Review Board (Approval number: SRB/SDC/PROSTHO-2206/23/176). The study aimed to evaluate the Shear Bond Strength (SBS) of indirect composite resin bonded to PEEK and PMMA before and after thermocycling.

Inclusion criteria:

- Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM)-milled PMMA and PEEK samples of standardised dimensions (1mm diameter);
- Indirect composite resin specimens;
- Bonding performed using standardised bonding agents and polymerisation techniques.

Exclusion criteria:

- Specimens with surface defects or irregularities after fabrication;
- Improperly polymerised composite resin specimens;
- Samples with visible contamination or bond failures before testing.

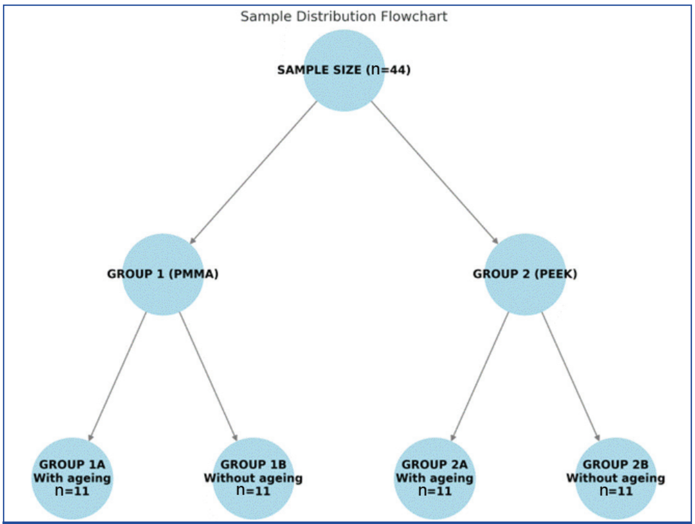
Sample size calculation: The sample size was determined using G*Power 3.1.9.3 for Mac OS X®, assuming a normal distribution. The effect size (dz=1.5004) and the sample size were estimated at an alpha of 0.05 and a power of 0.95 (1-β error probability) based on a prior study [6]. The estimated total sample size was 44. A total of 44 disc-shaped samples (22 PMMA and 22 PEEK) were included in the study. Each group was further subdivided into those subjected to thermocycling (Group 1A: PMMA with thermocycling, n=11; Group 2A: PEEK with thermocycling, n=11) and those not subjected to thermocycling (Group 1B: PMMA without thermocycling, n=11; Group 2B: PEEK without thermocycling, n=11) [Table/Fig-1,2].

Material group	Brand name	Composition	Manufacturer
Indirect composite	Shofu ceramage	73% zirconium silicate fillers (PFS-Progressive Fine Structured Fillers) evenly distributed in an organic polymer matrix	Shofu Dental, India
CAD/CAM milled PEEK	Intamsys PEEK	ZrO_2>97.7%+ HfO_2+ Y_2O_3 4.4%- 5.5%	Intamsys, Shanghai, China
CAD/CAM milled PMMA	Shandong Huge Dental Material Corporation	Polymethyl Methacrylate (PMMA) cross link	Shandong, China

[Table/Fig-1]: Details of materials, brand names, compositions, and manufacturers.

Study Procedure

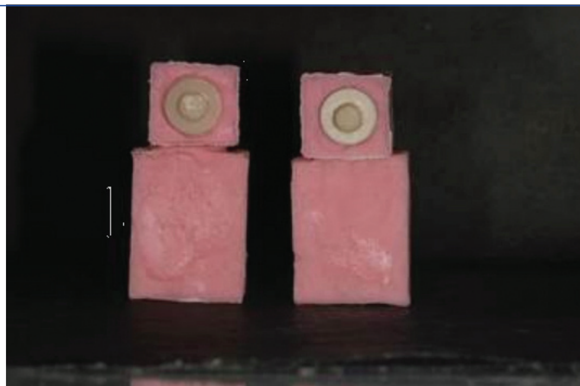
Sample preparation: The specimens were designed using Tinkercad software and Stereo Lithography (STL) files were obtained. The samples were fabricated from presintered PMMA and PEEK blocks using a 5-axis milling machine (IMES iCore, CORiTEC 350i milling machine®), yielding specimens with a diameter of 10 mm and a thickness of 2 mm. A clear silicone mould (Zhermack Elite Glass



[Table/Fig-2]: Descriptive flowchart of samples.

Silicon Transparent, Zhermack SpA, Italy) was prepared using one milled PMMA sample and this mould was used to create 44 indirect composite resin samples (Shofu Ceramage, Dentin A2 shade). To ensure uniformity and avoid void formation, a glass slab was placed over the mould before curing the composite resin in a Shofu Solidilite V curing unit (440-480 nm wavelength, intensity 1500 mW/cm²) for 40 seconds. Excess material was removed using abrasive papers of 100 and 600 grit.

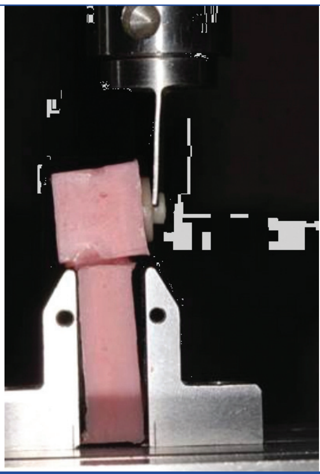
Bonding procedure: Acrylic resin blocks were fabricated using a customised iron mould measuring 30×15×12 mm. Self-cure acrylic resin was poured into the mould and the prepared PMMA and PEEK samples were embedded at the centre. The PMMA and PEEK groups underwent surface treatment using CeraResin Bond (CRB) (Shofu, Kyoto, Japan) before the application of the indirect composite resin. Bond I CRB was applied and air-dried for 10 seconds, followed by a second application of Bond II CRB, which was then light-cured. Following surface treatment, indirect composite resin (Shofu Ceramage, Dentin A2 shade) was systematically applied in layers to the PMMA specimens using a cylindrical metallic mould to ensure reproducibility and uniform thickness. Finally, the specimens were polymerised for 180 seconds before immersion in distilled water at 37°C for 24 hours [Table/Fig-3,4].



[Table/Fig-3]: Sample preparation with self-cure acrylic resin block with PEEK/PMMA samples and central composite disc.

Thermocycling: The samples were subjected to 1000 thermal cycles in water at temperatures ranging from 5°C to 60°C, with a dwell time of 30 seconds, using a thermocycler (Holmarc Thermocycler Model HO-THC-01). This process simulated approximately 1.2 months (36 days) of clinical ageing [21].

Shear Bond Strength (SBS) testing: The specimens were secured in a Universal Testing Machine (Instron E3000, Instron Corp, Massachusetts, USA) to measure SBS before and after thermocycling. A blade applied force at the interface between the indirect composite resin and the PMMA/PEEK discs at a crosshead



[Table/Fig-4]: Sample preparation for shear bond testing done under universal testing.

speed of 1 mm/min until failure occurred. The bond strength was recorded in megapascals (MPa).

STATISTICAL ANALYSIS

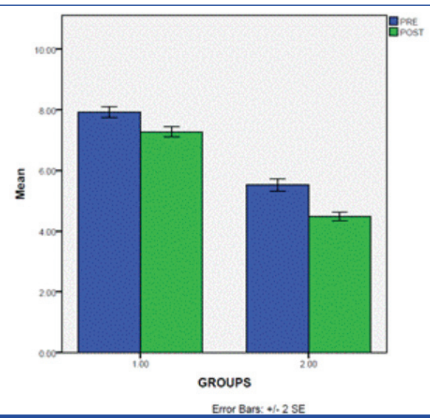
The data were analysed using Statistical Package For The Social Sciences (SPSS) software (version 23, IBM, Chicago, IL, USA). The Kolmogorov-Smirnov normality test confirmed normal data distribution, allowing the use of parametric independent sample t-tests for statistical analysis at a significance level of α=0.05.

RESULTS

The adhesion between the indirect composite resin and the PMMA material exhibited high bond strength, measuring 7.922±0.285 MPa before thermocycling and 7.270±0.2694 MPa after thermocycling. Descriptive statistics were employed, specifically the standard deviation and mean were used to compare values. The results showed a statistically significant difference (p-value <0.05). The indicates that the composite bonded to PEEK had a slightly lower SBS (p-value <0.05), with mean values of 5.520±0.341 and 4.486±0.232 is indicated in [Table/Fig-5]. Furthermore, the bond strength of the indirect composite resin to PMMA and PEEK decreased following thermocycling [Table/Fig-5,6]. The mean bond strength differed significantly between the groups.

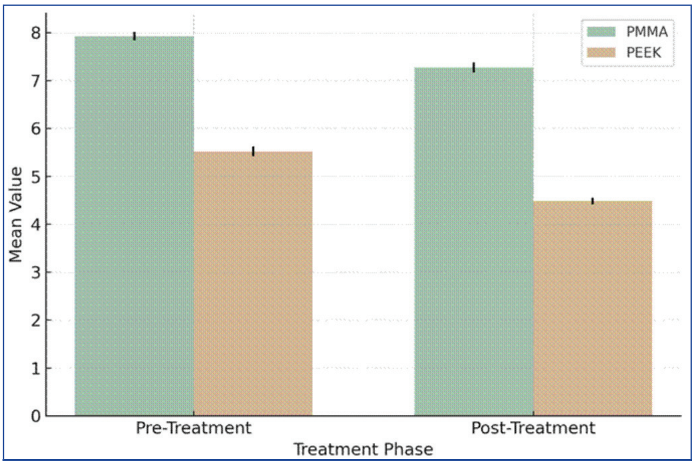
Groups		n	Mean (in MPa)	Std. deviation	Std. error mean	Sig.
PMMA	Pretreatment	11	7.9227	0.28570	0.08614	<0.01
	Post treatment	11	7.2709	0.26942	0.10284	
PEEK	Pretreatment	11	5.5200	0.34109	0.10284	<0.01
	Post treatment	11	4.4864	0.23265	0.07015	

[Table/Fig-5]: Mean values of pre- and post-thermocycling of Group 1 PMMA and Group 2 PEEK.



[Table/Fig-6]: The graph displays mean values of two groups, PMMA (I) and PEEK (II): Groups IA and IB, consisting of PMMA pre- and post-thermocycling, Groups IIA and IIB, consisting of PEEK pre- and post-thermocycling.

Intergroup comparison revealed a statistically significant difference between PMMA and PEEK in both Pretreatment (p-value <0.001) and post-treatment (p-value <0.001) conditions [Table/Fig-7].



[Table/Fig-7]: The graph displays an intergroup comparison of PEEK and PMMA in pre- and post-treatment.

DISCUSSION

The present study found a significant difference in bond strength between the indirect composite resin bonded to PMMA and PEEK, both before and after thermocycling (p-value <0.001). PMMA exhibited higher bond strength than PEEK in both conditions and thermocycling led to a reduction in adhesion for both materials. The null hypothesis was rejected in the present investigation since there was a statistically significant difference between the bond strengths of the indirect composite resin bonded to PMMA and PEEK, both pre- and post-aging.

The PMMA demonstrated significantly higher bond strength (7.92 MPa) compared to PEEK (5.52 MPa), which is consistent with previous research reporting superior adhesion of PMMA to composite resins due to its favourable surface characteristics and chemical compatibility with bonding agents [22]. Enhancing PMMA with reinforcing agents has been shown to improve its bond strength. Previous studies found that adding nano-fillers such as MgO significantly enhanced PMMA's adhesive properties. Specifically, PMMA containing 4% MgO exhibited a bond strength of 14.86 MPa, whereas unmodified PMMA had a bond strength of 8.19 MPa [23,24]. This suggests that modifying PMMA with reinforcing agents could further improve its application in composite veneering

In contrast, PEEK has gained popularity for dental prosthetic frameworks due to its high strength, biocompatibility and chemical resistance. However, its inherent gray opacity presents an aesthetic challenge, necessitating composite veneering for enhanced translucency and shade [25]. The lower bond strength of PEEK to composite resin observed in the present study is consistent with previous findings, which attribute this limitation to PEEK's low surface energy and resistance to chemical adhesion [26,27]. Several related studies have provided comprehensive insights into the effects of sulfuric acid solutions on PEEK polymers. These solutions effectively cleaved the aromatic polymer rings by targeting the carbonyl and ether groups, leading to enhanced surface polarity and wettability [28,29]. In contrast, PMMA achieves stronger adhesion through mechanical interlocking and chemical bonding, particularly when enhanced with reinforcing agents [30].

Thermocycling significantly reduced the bond strength of both PMMA and PEEK, highlighting the adverse effects of cyclic thermal stresses on adhesion. This reduction in bond strength has been attributed to hydrolytic degradation at the adhesive interface, leading to decreased stability over time [31,32]. Prior research has shown that PEEK, despite its chemical stability, experiences a decline in bond strength after thermocycling. For instance, one study reported that the bond strength of composite resin to PEEK decreased from 13.86 MPa to 13.46 MPa following thermocycling, indicating that PEEK undergoes changes under simulated ageing conditions [12]. Furthermore, reports suggest that PMMA experiences a more pronounced reduction in bond strength than PEEK due to its higher susceptibility to environmental degradation [33].

Given PEEK's low surface energy and chemical resistance, various surface treatments have been explored to enhance its bond strength to composite resin. These treatments include alumina sandblasting, tribochemical silica treatment and sulfuric acid etching [34]. Among these, sulfuric acid treatment has shown superior results in increasing bond strength, as it chemically modifies PEEK by cleaving its aromatic polymer rings, thereby improving surface wettability and adhesion [32,35]. In contrast, PMMA achieves stronger adhesion through mechanical interlocking and chemical bonding, particularly when enhanced with reinforcing agents [36].

The findings of the present study emphasise the importance of surface treatment in optimising bond strength, particularly for PEEK. The absence of surface modification in the present study may have contributed to its lower bond strength compared to PMMA. Future research should explore the effects of different conditioning techniques to improve PEEK's adhesive properties.

The present study underscores the significance of material selection in composite veneering applications. PMMA demonstrated superior bond strength with indirect composite resin, making it a more reliable choice for such applications. However, PEEK remains a promising alternative, especially if improved surface treatments are implemented to enhance its adhesion.

Limitation(s)

The present study was conducted in-vitro, which may not fully replicate oral conditions such as salivary enzymes and temperature fluctuations. Additionally, surface modifications were not performed, which could have affected the bond strength of PEEK. Future studies should explore optimised bonding strategies and long-term clinical outcomes.

CONCLUSION(S)

The present study found that thermocycling had a significant impact on the SBS of composite resin bonded to PMMA and PEEK. Thermocycling led to a reduction in adhesion for both materials. The comparative analysis showed that PMMA exhibited superior SBS with the indirect composite compared to PEEK. This highlights the importance of considering ageing conditions when evaluating adhesive properties. Future research should focus on optimising surface treatments for PEEK to enhance its bonding performance. Long-term clinical studies are needed to assess the durability of these bonds under functional loading. Additionally, exploring alternative adhesives and primers could provide further insights into improving adhesion in prosthetic applications.

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PARTICULARS OF CONTRIBUTORS:
1. Postgraduate Student, Department of Prosthodontics and Implantology, Saveetha Dental College, Chennai, Tamil Nadu, India.
2. Professor, Department of Prosthodontics and Implantology, Saveetha Dental College, Chennai, Tamil Nadu, India.

NAME, ADDRESS, E-MAIL ID OF THE CORRESPONDING AUTHOR:
Keerthi Sasanka,
Poonamallae High Road, Chennai, Tamil Nadu-600106, India.
E-mail: keerthis.sdc@saveetha.com

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